Method 1668B Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS
November 2008

This revision of Method 1668 (Method 1668B; the "Method") revises EPA Method 1668A to replace single-lab quality control (QC) acceptance criteria with interlaboratory criteria, and make other changes described below. Method 1668B was developed by the Office of Water's Office of Science and Technology (OST) for use in Clean Water Act (CWA) programs. Method 1668B is based on the results of an interlaboratory validation study, and a peer review of that study. Method 1668B, and the validation study report, *Method1668A Interlaboratory Validation Study Report* (EPA-821-08-021), are available at EPA's CWA methods website at www.epa.gov/waterscience/methods.

Method 1668B determines chlorinated biphenyl congeners in environmental samples by isotope dilution and internal standard high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS). The Method was developed for use in wastewater, surface water, soil, sediment, biosolids and tissue matrices. Other applications and matrices may be possible, which may or may not require modifications of sample preparation, chromatography, etc.

The detection limits and quantitation levels in this Method are usually dependent on the level of interferences and laboratory background levels rather than instrumental limitations. The estimated minimum levels of quantitation in Table 2 are concentrations at which a congener can be measured with laboratory contamination present. In water these values range from 10 to 500 parts per quadrillion (picograms per liter, pg/L). A laboratory may establish a lower reporting level for a congener, see Sect. 17.6.1.4.

This Method was prepared by Interface, Inc. and CSC Environmental Systems and Solutions under EPA Contract EP-C-06-085. Multi-lab (six labs for water and tissue, four for biosolids) data in Table 6 of this Method were provided by laboratories that participated in EPA's inter-laboratory validation of EPA Method 1668A. Previously, single-lab data for 1668A was developed by Axys Analytical Services, Ltd., Sidney, BC, Canada.

Summary of changes between EPA Method 1668A (8-20-03) and 1668B

- Based on the interlab validation study, single lab QC acceptance criteria are replaced with interlab criteria (Table 6.) A new footnote 1 to Table 6 references the EPA interlaboratory study report, and the other footnote numbers are incremented.

- Section 1.5, the performance-based discussion, describes additional flexibility to modify CWA methods that is allowed by 40 CFR Part 136.6.

- Section 2.5.2, now indicates that internal standards are the labeled congeners spiked into the sample.

- Section 2.5.3, now indicates that injection internal standards are labeled compounds

spiked into the extract.

- Section 5.4, is an added section on biohazards.
- Section 7.8, notes that Method 1668A part numbers are valid for Method 1668B.
- Section 8.1, allows use of alternate sample collection techniques, if documented.
- Section 8.2, adds that one liter, or a larger or smaller volume of sample, may be collected.
- Section 12.3, adds a note to indicate that SDS extraction may cause loss of some mono- through tri-chloro congeners.
- Section 12.5.6, states that a macro concentration device is to be used to concentrate extracts, and deletes the requirement for collection of the extract in a round-bottom flask because any macro concentration device may be used.
- Section 16.2, requires an expert spectrometrist to determine analyte presence when an interference precludes meeting the signal-to-noise requirement for dichloro-CB congeners.
- Section 21, references the validation studies, and that performance data are in the interlab validation study report.
- Reference 1, is updated to 2006 World Health Organization paper on toxicity equivalency factors.
- References 4 and 18, adds titles to the papers in these references.
- Reference 22, references the Method 1668A Interlaboratory Validation Study Report.
- Tables 2 and A-1, revise the elution order for congeners 107-109.
- Table 4, defines the solutions containing congeners 107, 108, and 109.
- Table 6, contains revised QC acceptance criteria for performance tests, and footnote 1 to Table 6 references the Method 1668A Interlaboratory Validation Study Report.
- Table 7, adds footnote 2 to require meeting the 10:1 signal-to-noise specification at the CS-2 calibration level.

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